

Application No. 10/611,743  
Amendment dated Feb. 17, 2005  
Reply to Office Action dated 11/17/04

### **REMARKS/ARGUMENTS**

In the Specification, the first paragraph on page 1 beginning with "The present invention ...herein by reference." has been amended to provide the patent number of the now issued parent patent application (Ser. No. 09/253,986; S-82,299) to this divisional patent application.

Claims 2-24 remain in this application. Claim 1 has been canceled. Claims 2-24 have been amended to recite a method for reducing the pore size of a ceramic inorganic membrane having a matrix of material particles of an inorganic compound including pores with pore walls therein comprising vapor treating the inorganic compound with a reactive vapor of a precursor inorganic compound which includes a reactive group that reacts with surface hydroxyls on said inorganic compound, and which reacts with water, so as to produce a reaction with the surface hydroxyls on the inorganic compound surface to thereby bond precursor molecules to the inorganic compound, and thereafter treating the inorganic compound surface with water vapor to convert the precursor inorganic compound into the corresponding inorganic compound and thereby producing a deposit of at least one monolayer of said precursor inorganic compound uniformly on the surface of said material particles.

In the Office Action dated November 17, 2004, original claim 5 was objected to because of the informalities that the word "and" at the end of line 4 should be deleted.

Original claims 1-3 and 5-8 were rejected under 35 U.S.C. 102(e) as being clearly anticipated by Levy et al. (5,789,024). More specifically, the Examiner stated that the method as claimed was disclosed in the Abstract, col. 1, lines 17-28, col. 7, lines 15-30, and col. 9, lines 8-35. He further noted that as to claim 8, the disclosure of heating in an evacuated vessel at col. 9 would accomplish drying of the membrane.

Original claims 1-10, 16, 17, and 22-24 were rejected under 35 U.S.C. 102(e) as being clearly anticipated by Funke et al. (6,051,517). The Examiner stated that the claimed process was disclosed at col. 4, lines 20-50, col. 4, lines 60-68, col. 7, lines 10-15, 25-30, and 60-65, col. 8, lines 30-45, col. 9, lines 1-3, and 35-50, col. 10, lines 21-55, and col. 11, lines 34-38. The Examiner remarked that the pore sizes disclosed in the examples after deposition of the layers are in the claimed ranges.

Original claims 1-3 and 5-8 were rejected under 35 U.S.C. 102(b) as being clearly anticipated by McMillan et al. (4,473,476). The Examiner stated that the process is explicitly disclosed at the abstract and Examples 1 and 2.

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Original claims 14 and 15 were rejected under 35 U.S.C. 103(a) as being unpatentable over either Funke et al. or McMillan et al. The Examiner stated that the above references do not explicitly disclose coating only one side of the membrane, such as by placing the membrane on a holder. He concluded that because the purpose of the membranes disclosed in the above references is to filter material which involves passing a medium through the membrane to allow some material to pass through based on the adjusted pore size and such passing through is only usually performed from a single direction through the filter to avoid dislodging trapped material filtered out by the membrane, it would have been obvious to coat only the inflow side of the filter to adjust the pore size thereof because that is the side at which filtration is performed and coating only one side would have clear advantages of saving process time and cost by coating only one side.

Original claims 11-13 and 18-21 were rejected under 35 U.S.C. 103(a) as being unpatentable over either Funke et al. in view of Levy et al. The Examiner stated that Funke does not explicitly disclose a gamma alumina or alumina membrane. He concluded that because Levy discloses that it is desirable to decrease the pore size of alumina membranes by depositing inorganic compounds thereon (col. 7, line 23), it would have been obvious to have coated an alumina membrane by the process of Funke with a reasonable expectation that doing so would successfully provide an alumina membrane having an adjusted pore size to tailor its filtration properties.

The objection to claim informalities in claim 5 has been corrected by deleting, as suggested by the Examiner, the word "and" appearing at the end of line 4.

Turning to the Examiner's rejection of claims 1-3, and 5-8 under 35 U.S. C. 102(e) as being clearly anticipated by Levy et al., this rejection is respectfully traversed. Levy et al. disclose (See Abstract, Col. 1, lines 10 – 28 as cited by the Examiner) a composite membrane having a high selectivity for a filtrate, e.g., nitrogen, which is formed utilizing opposing reactant geometry (i.e., a counterflow gas method) with at least two reactant gas streams to deposit via a low pressure chemical vapor deposition technique microporous film predominately of silicon oxide and optionally polysilicon, silicon carbide or silicon nitride within the walls of a mesoporous membrane substrate, thereby reducing the average pore size of the deposited film to an average pore size in the range of about 4 to about 8 Å.

Levy et al. at Col. 7, lines 15-30 (as cited by the Examiner), disclose a mesoporous skeleton substrate of a ceramic including  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Y}_2\text{O}_3$ , and  $\text{ZrO}_2$  or a borosilicate glass having a microporous film predominately of silicon dioxide with minor amounts of polysilicon, silicon carbide, and silicon nitride deposited on all or a portion of the pore and other surfaces of the substrate so that the region of the silicon film has an average pore size of less than about 8 Å. The resulting composite membrane is prepared by the therein disclosed self-terminating low pressure chemical vapor deposition process

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utilizing opposing reactant geometry (i.e., a counterflow gas method) to deposit the microporous film predominately of silicon oxide and optionally polysilicon, silicon carbide or silicon nitride within the walls of treated membrane.

Levy et al. at Col. 9, lines 8-35, cited by the Examiner, adds that the composite membrane is prepared by a low pressure chemical vapor deposition process wherein the mesoporous substrate is maintained at a temperature of from about 350 °C to about 650 °C and at a pressure in the range of from about 400m Torr to about 600m Torr.

The Levy et al. invention is bottomed on the discovery that the kinetic diameter of the molecules of the oxidizing reactant gas used in making their predominately silicon-based deposited films is critical to achieving the efficient selectivity and permeability of their composite membranes. For this discovery, the reactant gas stream requires the smallest reactant gas molecule to have a kinetic diameter intermediate in size between that of the matter retained by the filter and the filtrate passing there through; Levy et al. disclose an oxidizing reactant gas as nitrous oxide, N<sub>2</sub>O. This discovery is further taught as being critical to achieving their self-terminating deposition process and the pore size of the microporous film to achieve the efficient permeability and selectivity of the prepared composite membranes. (See Col. 11, lines 18-40 and also lines 41-56).

It may thus be seen that in order to achieve the self-terminating low pressure chemical vapor deposition process the critical elements of the Levy et al. process are (1) using opposing reactant geometry with at least two reactant gas streams to deposit a microporous film of predominately of silicon oxide and optionally polysilicon, silicon carbide or silicon nitride within the walls of a mesoporous membrane, and (2) the selection of an oxidizing reactant gas having a critical molecule diameter, i.e., one that is intermediate in size for the smallest reactant gas molecule between that of the matter retained by the filter and the filtrate passing there through, i.e., an oxide of nitrogen.

The PTO and courts have, historically, required for §102 anticipation that a single reference must teach (i.e., identically describe) each and every material element or step of the rejected claim. Here the Examiner has rejected claims 1-3 and 5-8 as being clearly anticipated by Levy et al., citing various sections of their Specification as disclosing Applicants' claimed method.

Applicants have now amended their claims by canceling claim 1 and amending claims 2, 3, and 5-8 to make each dependent from now amended claim 4, which has been written in independent form. All of the remaining claims, claims 2-24, have been amended to recite a method for reducing the pore size of a ceramic inorganic membrane having a matrix of material particles of an inorganic compound including pores with pore walls therein comprising vapor treating the inorganic compound with a reactive vapor of a precursor inorganic compound which includes a reactive group that reacts with surface hydroxyls

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on said inorganic compound, and which reacts with water, so as to produce a reaction with the surface hydroxyls on the inorganic compound surface to thereby bond precursor molecules to the inorganic compound, and thereafter treating the inorganic compound surface with water vapor to convert the precursor inorganic compound into the corresponding inorganic compound and thereby producing a deposit of at least one monolayer of said precursor inorganic compound uniformly on the surface of said material particles. Support for the addition of the word "ceramic" to the amended claims is found in Applicants' Specification at page 2, lines 29-32; page 4, lines 35-39; page 6, lines 20-22; and page 10, lines 14-17.

In contradistinction to the Levy et al. self-terminating low pressure chemical vapor deposition process for depositing a film of predominately silicon oxide (i.e., about 99% by weight) within the pores of the composite membrane, it is submitted that Applicants' invention is directed to a fundamentally different and distinct process for controlling the ultimate size of a fine-pored ceramic inorganic membrane having a matrix of material particles of an inorganic compound including pores with pore walls therein by depositing one monolayer at a time of an inorganic compound, e.g., metal oxides, metal carbides, and metal nitrides, uniformly on the surface of the particles which make up the pore walls of the pores of the matrix.

Applicants found, quite unexpectedly, that by vapor treating the ceramic inorganic membrane having a matrix of material particles of an inorganic compound with a reactive vapor of a precursor inorganic compound which includes a reactive group that reacts with surface hydroxyls on the inorganic compound and which reacts with water, the precursor molecules could be chemically bonded to the inorganic compound and after treating the inorganic compound surface with water vapor the precursor inorganic compound could be converted into the corresponding inorganic compound. In this way Applicants found that they could control the deposition of the precursor inorganic compound on the surface of the material particles one monolayer at a time. By this process Applicants can, unlike Levy et al., control the ultimate pore size of the inorganic membrane by repeating the deposition process to lay down one monolayer of the precursor inorganic compound at a time to achieve mean pore diameters below about 20 Å and even below 5 Å.

Additionally, Applicants' process is carried out in a temperature range of ambient temperature to about 300° C, whereas the Levy et al. low pressure process (i.e., in the range of 400 mTorr to about 600 mTorr) is carried out at a temperature in the range of about 350° C to 650° C for their chemical vapor deposition of a predominately silicon oxide film.

Lastly, Applicants' further step (as set forth in dependent claim 8) of drying the ceramic inorganic compound by heating and holding it at a temperature of 100° C to 200° C for one to two hours in an evacuated vessel was rejected by the Examiner on the Levy et al.

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disclosure at Col. 9. Viewing the Levy et al. disclosure at Col. 9 which shows maintaining the substrate at a temperature in the range of from about 350 °C to 650 °C it is seen that this further step of Applicants' claimed method is not found in the Levy et al. reference.

It is submitted that a fair reading of Levy et al. reference does not, as the Examiner contends, teach each and every material element or step of Applicants' rejected claims 1-3 and 5-8, as now amended.

Accordingly, it is respectfully requested that, in view of the claims as now amended and the above remarks, the rejection of Applicant's claims 1-3, and 5-8 under 35 U.S.C. 102(e) as being clearly anticipated by the cited Levy et al. reference, be withdrawn.

The Examiner also rejected original claims 1-10, 16, 17, and 22-24 under 35 U.S.C. 102(e) as being clearly anticipated by Funke et al. (6,051,517).

This rejection is also respectfully traversed. The Funke et al. reference is seen to be directed toward modified zeolite membranes or other crystalline membranes having a monomolecular layer deposited on the zeolite surface. That zeolite membranes have a fundamental different physical structure than the typical porous, ceramic, inorganic membranes (metal oxides, metal carbides and metal nitrides) was recognized by Applicants. (See Specification on page 3, lines 31-37) There, Applicants noted that the crystallographic structure of a zeolite defines the pore diameters in contrast to a ceramic membrane wherein the pores are the interstices between the particles.

Funke et al. confirm this fundamental difference in Col. 6, lines 5-19 where there is given a discussion of the zeolite membrane structure being a plurality of crystals, which are porous in the crystal lattice and later in lines 20-26 further noting that the pore openings are interconnected by a zig-zag system of straight channels with nearly circular cross-section.

This fundamental different structure between zeolite as disclosed by Funke et al. and the porous ceramic membranes used by Applicants translates into fundamental different approaches in solving the problem in the art of producing membranes that can effectively function as molecular sieves. This is best seen by reference to Fig. 2 and accompanying text in the Specification given at Col. 4, lines 61-63 and by reference to Figs. 2 and 5 and accompanying text at Col. 7, lines 45-48 of Funke et al. There it may be seen that the zeolite membrane has hydroxyl groups that are capable of reacting with volatile molecules only on the surface of the zeolite crystals, i.e., on the top, side and bottom or other surface (i.e., a gap or region between the juxtaposed faces of adjacent faces of the zeolite crystals). (See also Figs. 1 and 5 and text at Col. 6, lines 13-19)

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This is different from Applicants' porous ceramic inorganic membranes, which have a matrix of material particles including pores with pore walls therein with the hydroxyl groups being located on the walls of the membrane pores.

As noted above with regard to the Examiner's rejection of Applicants' claims on the Levy et al. reference, the PTO and courts have required for §102 anticipation that a single reference must teach (i.e., identically describe) each and every material element or step of the rejected claim. It is submitted that the rejection of Applicants' claimed invention as being clearly anticipated under 35 U.S.C. §102(e) by the Funke et al. patent fails to satisfy this requirement. None of the material elements or steps of Applicants' rejected claims are found or taught by Funke et al.

Accordingly, it is respectfully requested that, in view of the claims as now amended and the above remarks, the rejection of Applicant's claims 1-10, 16, 17, and 22-24 under 35 U.S.C. 102(e) as being clearly anticipated by the cited Funke et al. reference be withdrawn.

The Examiner also rejected claims 1-3 and 5-8 under 35 U.S.C. 102(b) as being clearly anticipated by McMillan et al., stating that the process is explicitly disclosed in the abstract and examples 1 and 2. This rejection is respectfully traversed.

McMillan et al. disclose a method of preparing a porous glass membrane for reverse osmosis by reacting a volatile metal chloride (e.g.,  $\text{TiO}_2$ ) with the chemically bonded hydroxyl groups on the surface of the glass thereby forming a metal chloride surface layer on the pores of the porous glass membrane and thereafter hydrolyzing the metal chloride surface layer by introducing water vapor into the  $\text{N}_2$  stream to produce a coating of metal oxide.

The starting porous glass membranes, which have a skeleton of silica, contain pores typically of 25 Å (radius). As noted therein if a salt solution and pure water are separated by a porous glass membrane containing pores of about 20 Å, the pure water will pass through the glass membrane to dilute the salt solution. By applying the method disclosed by McMillan et al. for coating the metal oxide on porous glass (beginning pore size of about 40 Å radius), suitable porous glass membranes were prepared having pores of between 18 to 20 Å (radius) that are useful in a reverse osmosis desalination process, i.e., causing water to leave a salt solution. McMillan et al. does not disclose or suggest a method for preparing porous ceramic inorganic membranes having extremely fine-pore structure having mean pore radius below about 5 Å as disclosed and claimed by Applicants.

Again, as noted above, the PTO and courts have required for §102 anticipation that a single reference must teach (i.e., identically describe) each and every material element or

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step of the rejected claim. Applicants' rejected claims, as amended, are not identically described in the McMillan et al. patent; neither, does the rejected claims, as amended, read literally on the single McMillan et al. cited reference. None of the material elements or steps of Applicants' rejected claims, as now amended, are found or taught by McMillan et al. It is submitted that the rejection of Applicants' claimed invention as being clearly anticipated under 35 U.S.C. §102(b) by the McMillan et al. patent also fails under this requirement.

Accordingly, it is respectfully requested that, in view of the claims as now amended and the above remarks, the rejection of Applicant's claims 1-3 and 5-8 under 35 U.S.C. 102(b) as being clearly anticipated by the cited McMillan et al. reference be withdrawn.

Claims 14 and 15 were rejected by the Examiner under 35 U.S.C. 103(a) as being unpatentable over either Funke et al. or McMillan et al. The Examiner stated that neither of the cited references explicitly discloses coating only one side of the membrane, such as by placing the membrane on a holder. He concludes that since the purpose of the membrane is to filter material which involves passing a medium through the membrane to allow some material to pass through, based on the adjusted pore size, and such passing is only usually performed from a single direction, it would have been obvious to coat only the inflow side of the filter to adjust the pore size. He notes that coating only one side would have the clear advantages of saving process time and cost by coating only one side as opposed to both sides.

This rejection is respectfully traversed. It is to be noted that claim 14, as now amended, is a dependent claim, depending from now independent claim 4 and claim 15 is a dependent claim from claim 14. As such these claims incorporate all of the limitations found in now independent claim 4.

In a §103(a) obviousness rejection the PTO has the initial burden of establishing a *prima facie* case and in order to meet this initial burden it must satisfy three requirements. First, the prior art relied upon, coupled with the knowledge generally available in the art at the time of the invention, must contain some suggestion or incentive that would have motivated the skilled artisan to modify or to combine references. *In re Fine*, 837 F.2d 1071, 1074, 5 U.S.P.Q. 2d 1596, 1598 (Fed. Cir. 1988); *In re Skinner*, 2 U.S.P.Q.2d 1788 (Bd. Pat. App. 1986).

Second, the proposed modification of the prior art must have had a reasonable expectation of success, determined from the vantage point of the skilled artisan at the time the invention was made. *Amgen, Inc. v. Chugai Pharmaceutical Co.*, 927 F.2d 1200, 1209, 18 U.S.P.Q. 2d 1016, 1023 (Fed. Cir.)

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Third, the prior art reference or combination of references must teach or suggest all the limitations of the claims. *In re Wilson*, 424 F.2d 1382, 1385, 165 U.S.P.Q. 494, 496 (C.C.P.A. 1970).

Additionally, the teachings or suggestions, as well as the expectation of success, must come from the prior art, not Applicants' disclosure. *In re Vaeck*, 947 F.2d 488, 493, 20 U.S.P.Q. 2d 1438, 1442 (Fed. Cir. 1991).

Neither the Funke et al. or McMillan et al. reference disclose, as correctly noted by the Examiner, coating only one side of the membrane. In fact the method disclosed in Funke et al. coats all surfaces of the zeolite crystals, i.e., on the top, side and bottom or other surface (gap or region between the juxtaposed faces of adjacent faces of the zeolite crystals). (See Figs.1 and 5 and text at Col. 6, lines 13-19 and lines 35-40). It is submitted that the method disclosed in the Funke et al. patent inherently coats all surfaces to modify the zeolite or other crystalline molecular sieves and therefore the requisite finding in the Funke et al. reference of a suggestion, incentive, or a reasonable expectation to coat only a single side of the membrane as found in the rejected claims is not found.

Similarly, the McMillan et al. reference, which deposits a surface layer of a metal oxide throughout the pores of a porous glass membrane, does not suggest, pose an incentive or hold out a reasonable expectation that the deposition could be deposited on only one side of the porous glass membrane, as disclosed and claimed by Applicants in amended dependent claims 14 and 15.

Absent a finding of these three requisites, all of which must come from either the Funke et al. or McMillan et al. references, the PTO has not established a *prima facie* case of obviousness and the rejection of claims 14 and 15 under 35 U.S.C. 103(a) on either of these two references must be withdrawn.

Lastly, the Examiner rejected claims 11-13 and 18-21 under 35 U.S.C. 103(a) as being unpatentable over either Funke et al. in view of Levy et al. The Examiner stated that Funke et al. did not explicitly disclose a gamma alumina or alumina membrane. He then, however, concluded that it would have been obvious in view of the Levy et al. disclosure that it is desirable to decrease the pore size of alumina membranes by depositing inorganic compounds thereon (Col. 7, line 23), to have coated an alumina membrane by the process of Funke et al. with a reasonable expectation that doing so would successfully provide an alumina membrane having an adjusted pore size to tailor its filtration properties.

This rejection is respectfully traversed. As noted herein, the Funke et al. disclosure is limited to modifying zeolite or other crystalline membranes for separation of materials on



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a molecular scale. As such, Funke et al. require hydroxyl groups located on the surfaces of the zeolite or other crystalline molecular sieves to carry out its method for depositing monomolecular layers on these surfaces of the zeolite crystals. As noted by the Examiner, there is no disclosure in Funke et al. for the use of a gamma or an alumina membrane.

As noted above, the PTO has the burden under a §103 obvious rejection to establish a *prima facie* case. This initial burden is met by satisfying the requirements of: (1) the prior art relied upon, coupled with the knowledge generally available in the art at the time of the invention, must contain some suggestion or incentive that would have motivated the skilled artisan to modify or to combine references, (2) the proposed modification of the prior art must have had a reasonable expectation of success, determined from the vantage point of the skilled artisan at the time the invention was made, and (3) the prior art reference or combination of references must teach or suggest all the limitations of the claims. Further, the teachings or suggestions, as well as the expectation of success, must come from the prior art, not Applicants' disclosure.

The Examiner correctly noted that Funke et al. does not disclose a gamma or alumina membrane. A careful review of the Funke et al. patent finds that it is devoid of any suggestion or incentive that would have motivated one to modify or combine references, as suggested by the Examiner, to utilize their process, which is limited to modifying zeolite or crystalline molecular sieve membranes, to coat a different type of membrane, i.e., an alumina membrane. Clearly, the Funke et al. patent does not teach or suggest all of the limitations of rejected claims 11-13, which depend from claim 4, as now amended, and claim 18-21, as now amended. Given this lack of disclosure of any suggestion or motivation to modify their process, it is submitted that the Examiner's assertion that such a modification to the contrary would be met with a reasonable expectation of success in reducing the pore size of a gamma or alumina membrane by Applicants' claimed process is equally without merit.

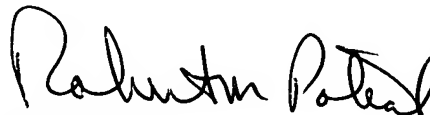
Absent a finding of these three requisites, all of which must come from the Funke et al. in view of Levy et al. reference, the PTO has not established a *prima facie* case of obviousness and the rejection of claims 11-13, and 18-21 under 35 U.S.C. 103(a) on the references must be withdrawn.

As the Examiner correctly noted, the above identified patent application currently names joint inventors. Applicants advise that the subject matter of the various claims was commonly owned at the time the invention covered therein was made, including amended claims 2-25, the remaining claims in the application.

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In view of the above amendments to claims 2-24 and remarks, this application is now believed to be in condition for allowance which action by the Examiner is respectfully requested.

Respectfully submitted,

A handwritten signature in black ink, appearing to read "Robert M. Poteat", written over a horizontal line.

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